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(54) Microbicidal Compositions

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A B S T R A C T

Microbicidal compositions which comprise a mixture of benzoic acid, methoxybenzoic acid, methylbenzoic acid, 2-furancarboxylic acid, ascorbic acid, pyruvic acid, sorbic acid or cyclohexanesulfamic acid or mixture thereof, with a C_{8-18} alkyl sulfate and/or sulfonate; or a mixture of tartaric acid or glycolic acid with benzoic acid or 2-furancarboxylic acid and a C_{8-18} alkyl sulfate and/or sulfonate. The compositions are useful for disinfecting a wide variety of surfaces and as hand and skin disinfectants, and, because they are toxicologically unobjectionable, they are particularly useful in the household, especially in the kitchen.

MICROBICIDAL COMPOSITIONS

BACKGROUND OF THE INVENTION

(a) Field of the Invention

The invention relates to microbicidal compositions which may be formulated as solids or liquids and, more particularly, to such compositions comprising as essential active ingredients certain organic acids and salts of aliphatic sulfates or aliphatic sulfonic acids.

(b) Information Disclosure Statement

Leading hygienists urgently express the need for fast-acting disinfectants, having a broad spectrum of microbicidal activity but the active components of which are toxicologically so innocuous that they can be employed in the area of food hygiene without any problem. Disinfection in the foodstuff industry presents a much more difficult problem than in the medical area since far fewer chemicals can be considered. To begin with, chemicals having strong odors or which are toxic are excluded from consideration. Among such chemicals are the aldehydes which, although possessing a broad spectrum of microbicidal activity, cannot be used due to their obnoxious odor as well as their ability to cause allergic reactions. Furthermore, the aldehydes are extremely incompatible with soap and their activity is reduced at temperatures below 18° C. Phenols, although numbering among the oldest of disinfectants, also are malodorous and toxic. As to the per-acids, halogens and halogen precursors, these agents develop unpleasant odors.

Other known disinfectants such as the quaternary ammonium salts have large gaps in their spectra of activity and are readily inactivated by protein contamination. Toxic effects are also encountered with guanidine derivatives while alcohols must be used at concentrations of 60% to 80% in order to obtain rapid disinfection. Such high concentrations of alcohols are to be avoided in spraying large surfaces because of the danger of explosion.

The urgent need for rapid-acting, toxicologically unobjectionable, broad-spectrum disinfectants for destroying agents which cause spoilage and pathogenic germs in the foodstuff industry extends also to the household kitchen, e.g., to dish washing, work areas, refrigerators and kitchen utensils. Obviously, such a disinfectant also would have advantages in the medical and sanitation areas.

U.S. Patent 2,393,866 to Wassell discloses metal tarnish remover compositions comprising water, abrasive particles, a polyoxyethylenediol, a tarnish-removing substance and a metal wetting agent. Among the classes of wetting agents disclosed are the salts of fatty alcohol sulfates. The tarnish-removing agents disclosed include organic acids. Among the various specific acids disclosed is tartaric acid.

U.S. Patent 3,083,166 to Harding discloses detergent compositions having improved detergency and brightening properties comprising non-soap anionic or nonionic detergents, a condensation product of formaldehyde and a carbocyclic aryl sulfonic acid, and an organic acid. Among the classes

anionic detergents disclosed are the alkali metal salts of higher alkylsulfonic acids and the alkali metal alkyl sulfates. Among the various specific organic acids disclosed are hydroxyacetic acid, lactic acid and tartaric acid.

U.S. Patent 3,141,821 to Compeau discloses compositions for local antisepsis comprising an anionic surface active sulfonate and a bacteriostatic compound at a pH of 2 to 4. Among the classes of surface active sulfonates disclosed are alkyl sulfonates having from 8 to 22 carbon atoms. The pH is regulated by the addition of an appropriate acid. Various specific acids are disclosed including tartaric acid and hydroxyacetic acid. No specific compositions comprising combinations of tartaric acid and/or hydroxyacetic acid and an alkyl sulfonate are exemplified.

U.S. Patent 3,650,964 to Sedllar et al. discloses low foam acid sanitizer compositions comprising anionic surfactants in acidic media. Among the classes of surfactants disclosed are the alkali metal salts of alkyl sulfates. Included in the various acids disclosed is hydroxyacetic acid. No specific compositions are exemplified which include a combination of hydroxyacetic acid or any organic acid with an alkali metal salt of an alkyl sulfate.

British Patent Specification No. 938,908 discloses a process for sterilizing and disinfecting fruit. A composition disclosed for comparative purposes comprises a 2% aqueous solution of the sodium salt of lauryl sulfate, hydroxyquinoline, salicylic acid, acetic acid and tartaric acid, and sodium propionate.

British Patent Specification No. 962,469 discloses anhydrous acid denture cleaner pastes comprising an anhydrous base and sulfonic acid. The pastes may contain other ingredients such as a surfactant. Several compositions are exemplified which contain, inter alia, sulfamic acid and sodium lauryl sulfate.

Australian Patent Specification No. 287,889 discloses anionic and non-ionic detergent baths containing a pH adjusting additive. Among the classes of anionic detergents disclosed are the alkyl sulfonates. The pH adjusting additive may be an organic or inorganic acid. Included among the specific organic acids disclosed is tartaric acid. No specific compositions containing an alkyl sulfonate and an acid are exemplified.

Chemical Abstracts 91, 44537p, discloses tabletted cleaning compositions for maxillary prosthesis comprising a phosphate or polyphosphate, a carbonate or bicarbonate, a salt of a weak organic acid, a detergent, an antimicrobial agent and a polyethyleneglycol. A specific composition exemplified contains, inter alia, tartaric acid and a detergent. The detergent is not specifically identified.

Chemical Abstracts 92, 182892x discloses sprayable cleaning compositions for removing soils and stains from hard surfaces containing sodium dihydrogen phosphate as well as oxalic acid, tartaric acid and/or citric acid. A specifically exemplified composition includes, inter alia, sodium stearyl sulfate and oxalic acid.

DETAILED DESCRIPTION INCLUSIVE OF THE PREFERRED EMBODIMENTS

It has now been found that the combination of alkyl sulfonates and/or alkyl sulfates with one or more organic acids described hereinafter displays an unexpectedly broad spectrum of microbicidal and virucidal activity in very low use concentrations at which the individual components of the combination do not exhibit any microbicidal activity. Such combinations possess no acute toxicity and are practically odorless.

Thus, for example, the minimum inhibitory concentration (MIC) for sorbic acid against Staphylococcus aureus is 0.7%, and against Escherichia coli it is 0.2%. However, with a mixture of 1 part of sorbic acid with 4 parts of a mixture of C_{8-18} alkyl sulfonates, killing is effected within 15 minutes at a sorbic acid concentration of only 0.0042% in the case of Staphylococcus aureus and only 0.0150% in the case of Escherichia coli. The MIC values for benzoic acid against Staphylococcus aureus and Escherichia coli are 0.1% and 0.125% respectively whereas with a mixture of 1 part benzoic acid with 4 parts of a mixture of C_{8-18} alkyl sulfonates, concentrations of benzoic acid of 0.0075% and 0.0300% suffice to kill Staphylococcus aureus and Escherichia coli respectively within 15 minutes.

By using suitable combinations of the acids employed in this invention, the spectrum of activity can be extended even to the allegedly resistant mildew fungi

such as Aspergillus niger, Aspergillus fumigatus and Penicillium expansum.

Thus in one aspect this invention provides a microbicidal composition comprising as essential components: (A) an organic acid selected from the group consisting of benzoic acid, benzoic acid substituted by a methoxy substituent or a methyl substituent, 2-furancarboxylic acid, ascorbic acid, pyruvic acid, sorbic acid and cyclohexanesulfamic acid and mixtures of said acids; and (B) a water-soluble salt of an alkyl sulfate or alkyl sulfonic acid wherein alkyl in each case contains from 8 to 18 carbon atoms.

In a second aspect this invention provides a microbicidal composition which comprises as essential components: (A) tartaric acid or glycolic acid; (B) benzoic acid or 2-furancarboxylic acid; and (C) a water-soluble salt of an alkyl sulfate or alkyl sulfonic acid wherein alkyl in each case contains from 8 to 18 carbon atoms.

The microbicidal compositions of the invention are active over a wide temperature range. Their activity at low temperatures is particularly advantageous for disinfecting cold rooms and refrigerators.

The alkyl sulfates and alkyl sulfonates employed in the invention are primary or secondary alkyl sulfates or sulfonates in which the alkyl group contains from 8 to 18 carbon atoms, preferably from 10 to 16 carbon atoms. The cation of the alkyl sulfates and alkyl sulfonates is alkali metal, e.g., sodium and potassium, ammonium, or substituted ammonium such as mono-, di- and triethanolammonium.

The sodium alkyl sulfates and alkyl sulfonates are preferred. Mixtures of alkyl sulfates and/or alkyl sulfonates can be employed.

The compositions of the invention can contain one or a mixture of two or more organic acids as follows: benzoic acid, benzoic acid substituted at the 2, 3 or 4-position by a methoxy or methyl substituent, 2-furancarboxylic acid, asorbic acid, pyruvic acid, sorbic acid and cyclohexane-sulfanic acid; or a mixture of tartaric and/or glycolic acid with benzoic acid or 2-furancarboxylic acid.

The weight ratio of alkyl sulfate or alkyl sulfonate to organic acid in the compositions of the invention can range from 50:1 to 1:50, preferably from 7:1 to 1:7, and more preferably from 9:1 to 1:1. The selection of a particular ratio will depend on the pK_a-value of the acid to be employed, as well as on the pH value desired in the use solution. The pH of the use solution should be between about 0.1 and 5.0. Optimal microbicidal activity is obtained in a pH range of 2 to 4.

The compositions of the invention can be formulated as solids, i.e., in granular or finely divided (powdery) form, which are prepared for use by dissolving in an aqueous medium. Alternatively they can be formulated as liquids in aqueous medium, either in a form ready for use or in the form of a concentrate which can be prepared for use by appropriate dilution in water. The concentrates generally will contain from about 20% to 40% by weight of

the active ingredients, i.e., the acid and alkyl sulfate or sulfonate. The concentration of active ingredients in the use solution should be sufficient to provide effective microbicidal activity as can be readily determined by one skilled in the art. The use solutions can be used safely and without danger in the household.

If the water solubility of a particular acid to be employed is limited, it is desirable to include an alcohol in the liquid forms of the compositions of the invention in order to aid in solubilizing the acid. Alcohols which can be used are, for example, ethyl alcohol, n-propyl alcohol, isopropyl alcohol and 1,2-propanediol. The alcohols also provide for faster drying of the compositions after application to a surface.

The compositions of the invention can optionally contain, in addition to the alkyl sulfates and sulfonates, other surfactants of the anionic, non-ionic and ampholytic types. Thus, if desired, anionic surfactants of the fatty alcohol ether sulfate type, e.g., sodium lauryl myristyl ether sulfate in combination with sodium chloride may be added in order to increase the viscosity of a use solution. If foam regulation of a use solution is desired, a non-ionic surfactant such as a fatty alcohol polyglycol ether with a suitable degree of ethoxylation can be employed.

Other optional ingredients which can be included in the compositions of the invention are corrosion inhibitors such as the phosphonic acids, low temperature stabilizers, enzymes, additional antimicrobial agents, perfumes, colorants,

solubilizers, pH-regulating agents, salts and, for the preparation of solid forms of the compositions, fillers such as sodium sulfate.

In view of their bactericidal, mycobactericidal, fungicidal, virucidal and desirable cleaning properties, the compositions of the inventions find utility not only in the home and with respect to foodstuffs in which case appropriate acids can be selected such as condiment acids, e.g., tartaric acid, and acids expressly permitted as additives in foodstuffs, but also are useful in appropriate concentrations as instrument, hand and skin disinfectants in hospitals, as well as in industry and agriculture. Their application in the field of dermatology also is possible.

For use as hand and skin disinfectants or in dermatology, the compositions can be formulated, by techniques standard in the art, as semi-solids, i.e., as paste-like, creamy or gel-like compositions. The alkyl sulfate or sulfonate employed in such compositions must be skin-compatible such as, for example, the triethanolamine salt of decyl sulfate. In formulating such compositions adjuvants such as organic and inorganic thickening agents, alcohols, fragrances and ointment bases can be employed.

Toxicological studies of the alkyl sulfates and sulfonates employed in this invention demonstrated an oral LD_{50} (mouse) of 2100 mg/kg which is physiologically unobjectionable.

The compositions of the invention are highly active, toxicologically unobjectionable disinfectants which

are particularly useful as microbicidal agents in the field of foodstuffs and in the household. They can be applied to the surfaces to be treated by well known procedures such as spraying, swabbing, immersion, etc.

The invention is illustrated by the following examples without, however, being limited thereto.

The compositions of the invention exemplified below were tested for antimicrobial activity in accordance with the methods of the DGHM (Richtlinien für die Prüfung chemischer Disinfektionsmittel der Deutsches Gesellschaft für Hygiene and Mikrobiologie) against two or more of the following bacteria, mold and fungi:

I. Staphylococcus aureus

II. Escherichia coli

III. Pseudomonas aeruginosa

IV. Proteus

V. Klebsiella

VI. Penicillium expansum

VII. Aspergillus niger

VIII. Aspergillus fumigatus

IX. Candida albicans

X. Tricophyton metagrophytes

The above-listed microorganisms are hereinafter identified by the above-designated Roman numerals.

Example 1

An aqueous composition, ready for use, was prepared containing the following ingredients:

Ingredient	Weight-Percent
L-(+)-Tartaric acid	0.5
Benzoic acid	1.0
Sodium alkyl sulfonate (mixture of C_{10-16})	2.0
Ethyl alcohol	20.0
Water (deionized)	76.5
	100%

pH 2.65

The antimicrobial and anti-viral test results for the composition of Example 1 were as follows:

1. Bactericidal Effect

(Suspension test; killing time in minutes)

Concentration (%)	Microorganism I	II	III	IV	V
50	2.5	2.5	2.5	2.5	2.5
10	2.5	2.5	2.5	2.5	2.5
3	2.5	15	2.5	2.5	15
1.5	2.5	>60	5	5	>60

2. Fungicidal Effect

(Suspension test; killing time in minutes)

Concentration (%)	Microorganism VI	VII	IX
100	2.5	2.5	2.5
50	2.5	2.5	2.5
25	15	5	2.5
10	>60	>60	15

3. Virucidal Effect v. poliomyelitis virus

Time (minutes)	Reduction of Titer, $\log_{10}$
5	>8
15	>8
30	>8
60	>8

4. Mycobacterium smegmatis was killed within 60 minutes.

5. Killing of bacteria on PVC and lacquered wood surfaces at room temprature and at 40°C.

The numbers listed in the time columns below represent the number of colonies of the microorganism, the first referring to the PVC surface and the second to the lacquered wood surface (e.g., 29/0 denotes 29 colonies on the PVC surface and 0 colonies on the lacquered wooden surface).

a) Room temperature

Microorganism	Time (minutes)			
	5	10	30	60
I	0/0	0/0	0/0	0/0
II	0/0	0/0	0/0	0/0
III	0/0	0/0	0/0	0/0
IV	0/0	0/0	0/0	0/0
V	29/0	0/0	0/0	0/0

b) 4°C

Microorganism	Time (minutes)			
	5	15	30	60
I	0/0	0/0	0/0	0/0
II	0/0	0/0	0/0	0/0
III	0/0	0/0	0/0	0/0
IV	0/0	0/0	0/0	0/0
V	100/17	0/0	0/0	0/0

6. Killing of fungi on PVC and lacquered wood surfaces at room temperature and 4°C.

The numbers listed in the time columns below have the same meaning as in paragraph 5) above.

a) Room temperature

Microorganism	Time (minutes)			
	5	15	30	60
VI	0/0	0/0	0/0	0/0
VII	0/0	0/0	0/0	0/0
IX	0/0	0/0	0/0	0/0

b) 4°C

Microorganism	Time (minutes)			
	5	15	30	60
VI	27/0	0/0	0/0	0/0
VII	0/0	0/0	0/0	0/0
IX	0/0	0/0	0/0	0/0

Example 2

A composition was prepared containing the following ingredients:

Ingredient	Weight-Percent
Sodium dodecyl sulfonate	20
L-(+)-Tartaric acid	5
Benzoic acid	5
Ethyl alcohol	15
Water (deionized)	55
	100%

The antimicrobial test results for the composition of Example 2 are given in Table 1.

Table 1

(Suspension test; killing time in minutes)

Concentration %	Microorganism I	III	IV	V	VII	IX	X
10	2.5	2.5	2.5	2.5	2.5	2.5	2.5
5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
1	2.5	2.5	2.5	2.5	2.5	2.5	2.5

Example 3

A solid (powdery) composition was prepared containing the following ingredients:

Ingredient	Weight-Percent
Sodium lauryl sulfonate	10
Sodium decyl sulfate	10
L-(+)-Tartaric acid	3
2-Furancarobxylic acid	5
Sodium sulfate	72
	100%

Example 4

A composition was prepared containing the following ingredients:

Ingredient	Weight-Percent
Sodium alkyl sulfonate (mixture of C_{10-16})	3.0
Cyclohexanesulfamic acid	0.9
Water (deionized)	96.1
	100%

pH 2.12

The antimicrobial test results for the composition of Example 4 are given in Table 2.

Table 2

(Suspension test; killing time in minutes)

	Concentration %	Microorganism I	II	III	IV	V	IX
	100	2.5	2.5	2.5	2.5	2.5	2.5
	25	2.5	2.5	2.5	2.5	2.5	5
	6.25	2.5	5	5	2.5	5	15
Control: Phenol	1%	30	30	2.5	15	5	60

Example 5

A composition was prepared containing the following ingredients:

Ingredient	Weight-Percent
Sodium alkyl sulfonate (mixture of C_{10-16})	3.00
1,2-Propanediol	5.00
L-(+)-Ascorbic acid	0.88
Water (deionized)	91.12
	100%

pH 2.95

The antimicrobial test results for the composition of Example 5 are given in Table 3.

Table 3

(Suspension test; killing time in minutes)

Concentration %	Microorganism I	II	IX
100	2.5	2.5	15
50	2.5	2.5	15
25	2.5	2.5	15
12.5	2.5	2.5	15
6.25	2.5	15	15
3.12	2.5	>60	30
Control: Phenol 1%	15	15	60

Example 6

A composition was prepared containing the following ingredients:

Ingredient	Weight-Percent
Sodium alkyl sulfonate (mixture of C_{10-16})	3.00
2-Furancarboxylic acid	0.56
1,2-Propanediol	5.00
Water (deionized)	91.44
	100%

pH 2.54

The antimicrobial test results for the composition of Example 6 are given in Table 4.

Table 4

(Suspension test; killing time in minutes)

	Concentration %	Microorganism I	II	IX
5	100	2.5	2.5	2.5
	12.5	2.5	2.5	2.5
	6.25	2.5	5	2.5
	3.12	2.5	5	2.5
	1.56	2.5	30	5
10	0.75	5	>30	>30
Control: Phenol	1%	30	30	30

Example 7

A composition was prepared containing the following ingredients:

Ingredient	Weight-Percent
Sodium alkyl sulfonate (C_{10-16})	3.00
Pyruvic acid	0.44
1,2-Propanediol	5.00
Water (deionized)	91.56
	100%

pH 2.18

The antimicrobial test results for the composition of Example 7 are given in Table 5.

Table 5

(Suspension test; killing time in minutes)

	Concentration %	Microorganism I	II	IX
	100	2.5	2.5	2.5
	50	2.5	2.5	5
	12.5	2.5	2.5	5
	6.25	2.5	5	15
	3.12	2.5	>60	15
Control: Phenol	1%	15	30	60

Example 8

A composition was prepared containing the following ingredients:

Ingredient	Weight-Percent
Monoethanolamine dodecyl sulfate	3.00
Glycolic acid	0.38
Benzoic acid	1.00
Ethyl alcohol	20.00
Water (deionized)	75.62
	100%

pH 2.6

The antimicrobial test results for the composition of Example 8 are given in Table 6.

Table 6

(Suspension test; killing time in minutes)

Concentration %	Microorganism VI	Microorganism VII
100	2.5	2.5
50	2.5	5
25	30	15

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A microbicidal composition comprising as essential components:

(A) an organic acid selected from the group consisting of benzoic acid, benzoic acid substituted by a methoxy substituent or a methyl substituent, 2-furancarboxylic acid, ascorbic acid, pyruvic acid, sorbic acid and cyclohexanesulfamic acid and mixtures of said acids; and

(B) a water-soluble salt of an alkyl sulfate or an alkyl sulfonic acid, wherein alkyl in each case contains from 8 to 18 carbon atoms, or mixtures of said salts; wherein the ratio of component (B) to component (A) is from 50:1 to 1:50.

2. The composition according to claim 1, wherein the ratio of component (B) to component (A) is from 7:1 to 1:1.

3. The composition according to claim 1, wherein the salt of the alkyl sulfate and alkyl sulfonic acid is an alkali metal salt, the ammonium salt or a substituted ammonium salt.

4. The composition according to claim 3, wherein component (B) is an alkali metal salt of an alkyl sulfonate.

5. The composition according to claim 4, wherein component (B) is a mixture of sodium alkyl sulfonates having from 10 to 16 carbon atoms.

6. The composition according to claim 5, wherein component (A) is cyclohexanesulfamic acid; and the ratio of component (B) to component (A) is about 3.3:1.

7. The composition according to claim 5, wherein component (A) is L-(+)-ascorbic acid; and the ratio of component (B) to component (A) is about 3.4:1.

8. The composition according to claim 5, wherein component (A) is 2-furancarboxylic acid; and the ratio of component (B) to component (A) is about 5.4:1.

9. The composition according to claim 5, wherein

component (A) is pyruvic acid; and the ratio of component (B) to component (A) is about 6.8:1.

10. A microbicidal composition which comprises as essential components:

(A) tartaric acid or glycolic acid

(B) benzoic acid or ~~2-furancarbocylic~~ 2-furancarboxylic acid; and

(C) a water-soluble salt of an alkyl sulfate or alkyl sulfonic acid wherein alkyl in each case contains from 8 to 18 carbon atoms, or mixtures of said salts;

wherein the ratio of component (C) to combined components (A) and (B) is from 50:1 to 1:50.

11. The composition according to claim 10, wherein the ratio of component (C) to combined components (A) and (B) is from 7:1 to 1:1.

12. The composition according to claim 10, wherein the salt of the alkyl sulfate and alkyl sulfonic acid is an alkali metal salt, the ammonium salt or a substituted ammonium salt.

13. The composition according to claim 12, wherein component (C) is sodium alkyl sulfate, monoethanolamine alkyl sulfate or sodium alkyl sulfonate or mixtures thereof, wherein alkyl in each case has from 10 to 16 carbon atoms.

14. The composition according to claim 13, wherein component (A) is L-(+)-tartaric acid; component (B) is benzoic acid; and component (C) is a mixture of sodium alkyl sulfonates having from 10 to 16 carbon atoms; and the ratio of component (C) to combined components (A) and (B) is about 1.3:1.

15. The composition according to claim 13, wherein component (A) is L-(+)-tartaric acid; component (B) is benzoic acid; and component (C) is sodium dodecyl sulfonate; and the ratio of component (C) to combined components (A) and (B) is about 2:1.

16. The composition of claim 13, wherein component (A) is glycolic acid; component (B) is benzoic acid; and component (C) is monoethanolamine dodecyl sulfate; and the

ratio of component (C) to combined components (A) and (B) is about 2.2:1.

17. The composition according to claim 13, wherein component (A) is L-(+)-tartaric acid; component (B) is 2-furancarboxylic acid; and component (C) is a mixture of sodium lauryl sulfonate and sodium decyl sulfate; and the ratio of component (C) to combined compounds (A) and (B) is about 2.5:1.

SUBSTITUTE

REMPLACEMENT

SECTION is not Present

Cette Section est Absente